

Name I. C. LEWIS / T. PIRRO

Notebook Number 195-129

Subject Chemistry of Biomaterials, New Biologics & Implants

Dates From _____ To _____

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Purpose:

To obtain initial wt and dimensions prior to vacuum impregnation w/ a "T-143 TYPE" Resin

Mat'l:

c/c Composite via BP Process # 4-1: Rec'd from P. S. Rocky 4/30/01. From 1st Lawrenceburg Trial. Block 4-1 ~ 1.5 x 3.0 x 7.0 inches. From 4th block in the series. Made w/ 0.25" long pitch fibers and Kevlar 155 pitch. Wool Ratio 85/15 + 5 with sulfur based on the pitch weight.

Procedure:

Ultrasonic washed in deionized H₂O 3x, 5min intervals \Rightarrow Vacuum dried overnight at ~150°C to 2.4mm pressure.

Unloaded & cooled in desiccator, 5/8/01. Weighed & dimensioned 5/11/01 \Rightarrow loaded into oven at ~150°C, 50 SCFH argon purged until loaded in to VI unit.

Weight & Dimension: (5/11/01)

wt (g)	L ₁	L ₂	L ₃	Ave LEN.	w ₁	w ₂	w ₃	Ave WIDTH	H ₁	H ₂	H ₃	Ave Ht.	Vol. (cc)
1138.84	191.91	192.27	192.02	192.07	83.78	83.36	82.32	83.15	47.21	46.71	46.65	46.86	748.35

$$\Rightarrow \text{Density}_{c/c} = 1.522 \text{ g/cc}$$

Comments:

composite contains severe defects, such as fissures and splinters. One area appears to contain iron oxide.

VI and Cure: Ref. 19-124-~~55~~ 55256

Performed and Recorded by: 

Directed by: J. (lin)

Read and Understood by:

Date

Date

Date

Subject Preparation of 2nd 900 ml Aliquots of Solids by Volume GP-5432/Furfural (decant) 53
Cross-Reference (if any)

Purpose:

To impregnate 195-129-52 w/la "T-143 Type" resin.

Materials:

GP-5432: Lot # 19588. Rec'd from Georgia-Pac. Inc 9/24/90. LIMS # A96-03635. Stored in Freezer. Anal. Mod MCC (3) = 48.6 ($\sigma = 0.37, n = 3$), Brookfield Viscosity = 157.3 cP at 71.0°F, PDSC Data: Ref. 195-105-49, TGA Yield (900°C) = 47.5%, Current Visc = 283.5 cP at 68.7°F
Furfural: Reagent Grade (Fisher). Rec'd 1/15/01. 2nd Aliquot used 60 ml of 2nd 500 ml rec'd 5/15/01 \Rightarrow Balance = 940 ml.

Preparation 1st Aliquot: (5/14/01) - 1L Erlenmeyer Flask. 450 ml GP-5432 + 450 ml Furfural

FLASK (etc) + GP-5432 (3) = 996.3	FLASK (etc) + FURFURAL (3) = 1516.0
FLASK (etc) (3) = <u>455.9</u>	FLASK (etc) (3) = <u>996.3</u>
450 ml GP-5432 (3) = 540.4g	450 ml FURFURAL (3) = 519.7g \Rightarrow 50.98% GP-5432 by wt

Comments:

Mix stirred w/o external heat for 15 min after combining \Rightarrow Transfer to 32 oz glass jar.

Characterization of the 1st Aliquot: (5/15/01)

Brookfield (LVT) AT Viscosity:

Viscosity = 18.3 cP at 71.8°F Spindle #1, 60 RPM, Factor = 1

Spec. Gr. Gravity at RT:

S.G. = 1.190 at 71.8°F

Preparation of 2nd Aliquot: (5/15/01) - 1L Erlenmeyer Flask. 450 ml GP-5432, 450 ml Furfural

FLASK (etc) + GP-5432 (3) = 995.0	FLASK (etc) + FURFURAL (3) = 1512.6
FLASK (etc) (3) = <u>455.9</u>	FLASK (etc) (3) = <u>995.0</u>
GP-5432 (3) = 539.1g	FURFURAL (3) = 517.6g \Rightarrow 51.02% GP-5432 by wt

Solutions combined 5/16/01. Label as 195-129-53 1st Use: Ref. 195-129-55

% Mod MCC of Aliquot #1: Ref. 195-129-54

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Directed by: 

Read and Understood by:

Date

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Date

Material:

195-129-53, 1st Aliquot: 50/50 by volume, 51.0/49.0 by weight 68-5432/FU
Prep. 5/14/01. Viscosity = 18.2 cP at 31.8°C, SG at 1 = 1.190 at 712. Mod MCC Determinations: (5/15/01) - ~1.5g sample size. Cured 1hr at 144°C, 5.0 sec.

$$\begin{aligned}
 1) \text{CRUC} + \text{CHIPS} + \text{SAMPLE WT} &= 19.1507 & \text{CRUC} + \text{CHIPS} + \text{SAMPLE}_{(144)} &= 18.3997g \Rightarrow \text{Yield}_{(144)} = 50.5\% \\
 \text{CRUC} + \text{CHIPS WT} &= \underline{17.6308} & \text{CRUC} + \text{CHIPS} + \text{SAMPLE}_{(MCC)} &= 18.1502g \Rightarrow \text{Yield}_{(MCC)} = 67.5\% \\
 \text{SAMPLE WT} &= 1.5199g
 \end{aligned}$$

$$\text{TOTAL YIELD} = (0.5059 \times 0.6755) \times 100 = \underline{34.17\%}$$

$$\begin{aligned}
 2) \text{CRUC} + \text{CHIPS} + \text{SAMPLE WT} &= 18.9635 & \text{CRUC} + \text{CHIPS} + \text{SAMPLE}_{(144)} &= 18.2000g \Rightarrow \text{Yield}_{(144)} = 49.20\% \\
 \text{CRUC} + \text{CHIPS WT} &= \underline{17.4606} & \text{CRUC} + \text{CHIPS} + \text{SAMPLE}_{(MCC)} &= 17.9770g \Rightarrow \text{Yield}_{(MCC)} = 69.84\% \\
 \text{SAMPLE WT} &= 1.5029g
 \end{aligned}$$

$$\text{TOTAL YIELD} = (0.4920 \times 0.6984) \times 100 = \underline{34.36\%}$$

$$\begin{aligned}
 3) \text{CRUC} + \text{CHIPS} + \text{SAMPLE WT} &= 18.3929 & \text{CRUC} + \text{CHIPS} + \text{SAMPLE}_{(144)} &= 17.6460g \Rightarrow \text{Yield}_{(144)} = 50.5\% \\
 \text{CRUC} + \text{CHIPS WT} &= \underline{16.8837} & \text{CRUC} + \text{CHIPS} + \text{SAMPLE}_{(MCC)} &= 17.4073g \Rightarrow \text{Yield}_{(MCC)} = 68.6\% \\
 \text{SAMPLE WT} &= 1.5092g
 \end{aligned}$$

$$\text{TOTAL YIELD} = (0.5051 \times 0.6869) \times 100 = \underline{34.69\%}$$

Comments:

All samples were held at temperature after cure at ~144°C.

$$\text{Ave Yield}_{(144)} = 50.1\%, \sigma = 0.78, n = 3$$

$$\text{Ave Yield}_{(MCC)} = 68.7\%, \sigma = 1.15, n = 3$$

$$\text{Ave Mod MCC} = 34.4\%, \sigma = 0.26, n = 3$$

Performed and Recorded by:

Directed by:

Read and Understood by:

Date:

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Subject VI + Cure to $\sim 200^\circ\text{C}$ of 195-129-52 #4-1 w/ 195-129-53
Cross-Reference (if any)

(c/c COMP BP) 55

Purpose:

To density c/c composite w/ phenol-resin/furfural blends. To verify max vol% pickup w/ pitches in the PC.

Materials:

- 1) c/c composite: 195-129-52 #4-1 (c/c composite via BP process. From 4th Block in the 12th Lawrenceburg Trial. 0.25" long pitch fibers + Reilly 155 Pitch. Load ratio 85/15 + 5 wt% sulfur based on the pitch wt.) $\text{Vis}_{(2)} = 1178.84\text{g}$, $\text{Vol}_{(2)} = 748.383\text{cc}$, $\text{Dens}_{(2)} = 1.522\text{g/cc}$
- 2) Impregnant: 195-129-53 50/50 by volume 60-5432 + furfural. Prep. 5/14/01.
 $\text{Vis}_{(1)} = (17.4)\text{cP}$ at 74.5°F , $\text{SG}_{(1)} = (1.188)$ at 74.5°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 + 16

Pump-down Data: (5/14-16/01)

DATE	TIME	PRESS (mTorr)	Comments
5/14	13:30	15	load c/c composite from oven (i.e. $\sim 150^\circ\text{C}$, Atm. Pressure).
"	13:40	"	
"	16:05	16	
5/15	7:20	14	
"	16:00	16	
5/16	7:25	13	
"	8:05	15	change traps w/ dryice-acetone
"	8:50	10	LDR
"	11:45	10	Begin VI

Impregnation Dates: (5/16 + 17/01) - LDR w/ traps changed

LDR: Initial = 10 millitorr $\text{Vis}_{(1)} = 17.4\text{cP}$ at 74.5°F
 5 min = 21 " $\text{SG}_{(1)} = (1.188)$ at 74.5°F
 10 min = 27 "
 15 min = 34 "

Drop Time = 11:45 (10 mTorr)
 Unload Time = 8:45 (5/17/01)
 Held at atmospheric pressure for
 $\sim 21\text{ hrs.}$

Comments:

570 ml of impregnant in the 500 ml cylinder + funnel.

cont'd next page

Performed and Recorded by: 

Directed by: J (Lew)

Read and Understood by:

Date

Date

Date

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Subject

V.I and Cure (w/350°C) Data of 195-129-52 #4-1 w/195-129-53

(C/C COMP B1)

Cross-Reference (if any)

Post Impregnation Data: (5/17/01)

Wt_(CPR1-1) = 1320.11g \Rightarrow Wt Pickup = 181.27g \Rightarrow Wt/o Pickup = 15.92, Vol/o Pickup = 20.40
 At Pan + Screen (C1) = 181.4g

Curing Data: (5/17/01) - Cure in small (A) Pan w/an s.s. screen to determine amount of run-out.

	TIME	EVEN SET	EVEN TEMP	Comments
*	8:45	48	158	Load into oven. Purge w/argon at 5.0 SCFH (AIR)
	9:15	"	156	Wet resin on all visible surfaces. Condensation on oven door.
	9:30	"	154	Impregnant boiling on surfaces. Runout on screen in the pan.
	10:05	"	158	Boiling has ceased. Resin is likely cured.
-(2)	10:40	"	156	Unload to desiccator. Set oven at "82" (30%). Cool etc. w/pan to 70.5g.
	-	-	-	Wt = 1232.35g \Rightarrow Wt Pickup = 93.51g \Rightarrow Wt/o Pickup = <u>8.21</u> (Yd = <u>51.6%</u>).
	-	-	-	Pan + Screen + Run-out = 188.4g \Rightarrow Cured Run-out = <u>7.9g</u>
*	11:55	82	245	Load over (180° Rotation " to bottom).
-(3)	13:55	"	248	Power off. Allow oven to cool to ~150°C \Rightarrow unload to desiccator
	15:25	OFF	156	Unload to desiccator. Cool overnight \Rightarrow weigh following morning

Post Curing Data: (5/18/01)

Pan + Screen + Cured Run-out (C1) = 188.3g \Rightarrow Cured Run-out = 6.9g

Wt_(C1) = 1212.74g \Rightarrow Wt Pickup = 73.9g \Rightarrow Wt/o Pickup = 6.49 (Yd = 40.8%)

\Rightarrow Impregnant Yield (including the run-out) = 44.6% \Rightarrow Density (C1) = 1.620 g/cc

Label 195-129-56, Give to P. Smocky 5/21/01

Rebar Data: Ref. 195-129-76

Performed and Recorded by:

Directed by: C. Lin

Read and Understood by:

Date

Date

Date

Material:

195-129-56: c/c Composite via BP process from the 4th Block in the 1st Lawrenceburg trial. 0.25" long pitch fiber + Reilley 155 Pitch. Land Ratio 85/15 + 5 wt% sulfur based on pitch wt. Initial Data: Ret. 195-129-52 #4-1, VI + Cure Data: Ret. 195-129-55 + 56. Rec'd from P. Sirocky 6/20/01.

Rebake Cycle (per P. Sirocky):

10°C/hr to 900°C, 2 hr hold. Block was warm when received. Cooled in desiccator \Rightarrow weighed + dimensioned.

Rebake Data: (6/20/01)

wt (g)	L ₁	L ₂	L ₃	AVE. LEN.	W ₁	W ₂	W ₃	AVE. WIDTH	H ₁	H ₂	H ₃	AVE. Ht.	Vol (cc)
1179.43	192.10	192.32	192.37	192.26	83.98	83.62	82.66	83.49	46.55	46.42	46.60	46.59	747.836

Rebake Density = 1.577 g/cc $\Rightarrow \Delta = 0.055$ g/cc over "green" density.

Wt% (green thru bake) = 3.56%

Vol% (green thru bake) = -0.07% (essentially no change)

Dal% (green thru bake) = 3.61%

Wt% (cure thru bake) = -2.75%

Returned to P. Sirocky 6/20/01. Data communicated via e-mail to D. Hoang, P. Sirocky, & E. Pincost.

Comments:

* Impregnant % wt Yield from VI thru Rebake = $(40.59/181.27) \times 100 = 22.4\%$

Note:

% Mod MCC = 34.4 ($\sigma = 0.26, n = 3$) for the impregnant \Rightarrow The difference is due to run-out and/or weight loss from the pitch binder because it hadn't been to ~900°C yet.

Performed and Recorded by: 

Directed by: J. Kim

Read and Understood by:

Date

Date

Date

Subject Initial Data of c/c Composites via BP Process (3rd Trial)

Cross-Reference (if any)

(c/c Comp)

Purpose:

To obtain initial weights and dimensions prior to vacuum impregnation with Silicon (IV) oxide colloidal dispersion for insitu conversion to SiC.

Materials:

c/c Composite via BP process. Rec'd From P. Sirocky 7/16/01. Two bricks section in ~ half. from the 3rd Lawrenceburg trial. Made w/ 0.25" long K-2235E pitch fibers and Reilley 155 pitch. Load Ratio 75/25 w/o sulfur.

Procedure:

Ultrasonic washed in deionized H₂O 3x, 5 min intervals. \Rightarrow vacuum-dried on at ~166°C, 0.1 mm pressure.

Air cooled, will not fit in desiccator, \Rightarrow weighed & dimensioned.

Initial Data: (7/19/01)

File Path = c:\Program Files\Excel\BP C-C Composites\Initial.xls Sheet = BP III

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-III-1 and BP-III-2. Rec'd 7/16/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/18/01. Dimensions were obtained with a Starrett No. 122-12 vernier caliper. Hot vacuum dried at 166 °C to 0.1 mm pressure from 7/18 to 7/19/01. Weights obtained on Mettler PN 2210 balance on 7/19/01.

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-1-A	916.19	8.173	8.155	8.110	8.146	3.261	3.305	3.304	3.290	1.333	1.302	1.288	1.308	574.297	1.595
3-1-B	823.65	8.196	8.200	8.197	8.198	3.296	3.271	3.248	3.272	1.253	1.273	1.259	1.262	554.502	1.485
AVERAGE =															1.540
STND. DEV. =															0.0777

Sample I.D.	Weight (g)	L1 (in.)	L2 (in.)	L3 (in.)	Ave. Length (in.)	W1 (in.)	W2 (in.)	W3 (in.)	Ave. Width (in.)	H1 (in.)	H2 (in.)	H3 (in.)	Ave. Height (in.)	Vol. (cc)	Dens. (g/cc)
3-2-A	867.13	8.883	8.863	8.837	8.861	3.226	3.265	3.298	3.263	1.183	1.170	1.169	1.174	556.247	1.559
3-2-B	861.58	8.913	8.905	8.896	8.905	3.263	3.281	3.292	3.279	1.196	1.207	1.180	1.194	571.400	1.508
AVERAGE =															1.533
STND. DEV. =															0.0361

Dimensioned: 07/19/01

Hot Vac. Dried: 07/18-19/01

Weighed: 07/19/01

N.B. Ref. No. : 195-129-86

#	Impregnant	NB Reference
3-1-A	Silicon (IV) Oxide Colloidal Dispersion	195-129-88 & 89
3-1-B	"	195-129-92 & 93
3-2-A	"	2VJ 195-129-94 & 95
3-2-B	"	" 195-129-96 & 97

Performed and Recorded by: 

Directed by: 

Read and Understood by:

Date:

Date:

Date:

Subject VI of 195-129-86 #3-1-A w/ Silicon (III) Oxide Colloidal Dispersion C/c com
Cross-Reference (if any)

Purpose:

To investigate in situ conversion of carbon to SiC in a c/c composite

Materials:

- 1) c/c Composite: 195-129-86 #3-1-A: (c/c composite via BP process from the 1st B10 of the 3rd Lawrenceburg trial, 0.25" long K-2235E pitch Fibers and Reiley 155 p. Load ratio 75/25. No sulfur, Wtcs = 916.19g, Volcs = 574.297cc, Dencs = 1.595g
- 2) Impregnant: Silicon (III) oxide, 30% in H₂O, colloidal dispersion, (Alfa-Aesar), Lot # A04K09, 0.01 μ m particles, in liquid, S.A. = 320 m²/g. Density = Viscos 7.7 cps at 82.1°F, S.G. (s) = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15 + 16 Use teflon support and Pyrex pan for drying.

Pump-down Data: (7/20-23/01)

DATE	TIME	PRESS (mTorr)	Comments
7/20	12:45	18	Load c/c composite from cold, dry over.
"	13:00	"	Begin pump-down.
"	14:00	44	
"	15:35	28	
7/23	7:20	21	
"	8:00	"	Charge traps w/ dry ice-acetone.
"	9:05	14	LDR
"	11:50	13	Begin VI

Impregnation Data: (7/23 + 24/01) - LDR w/ traps charged

LDR: Initial = 14 mTorr Viscos = 7.7 cps at 82.1°F Drop Time = 11:50 (03 m)
 5 min = 27 " S.G. (s) = 1.216 at 82.1°F Unload Time = 8:00 (7/24)
 10 min = 38 " Held at atmospheric press.
 15 min = 49 " for ~ 20 1/4 hrs.

Comments:

500 cc cylindrical Funnel is full. Return impregnant to ② 1 qt. poly-bot

Post Impregnation Data: (7/24/01)

Wt (pvi-1) = 1029.86g \Rightarrow Wt Pickup = 113.67g \Rightarrow Wt/Lg Pickup = 12.41 Vol/Lg Pickup = 16.

Performed and Recorded by: [Signature]

Date: [Blank]

Directed by: [Signature]

Date

Read and Understood by:

Date

Subject VI of 19J-129-86 #3-1-A w/ siliceous (III) oxide Colloidal Dispersion (c/c COMPAP) 89
 Cross-Reference (if any)

Drying Data: (7/24 & 25/01) - Over set "29" (50%). Argon purge = 5.0 SCFH (AIR)

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:10	108	Atm	Load over; # to top. Over set " <u>29</u> " (50%). Purge w/ argon at 5.0 SCFH (AIR)
(3)	11:10	106	"	Unload over. Set over at " <u>38</u> " (50%). Weigh brick hot (2. doesn't fit in desiccator) $Wt = 989.00g \Rightarrow Wt. Pickup = 72.81g \Rightarrow Wt/c Pickup = 7.95 (Yd = 6.41\%)$
	-	-	-	
*	11:30	125	Atm	Load over; # to bottom. Over set at " <u>38</u> " (50%). Purge w/ argon at 5.0 SCFH (AIR)
(2)	13:30	130	"	Unload over. Set over at " <u>46</u> " (50%). Weigh brick hot. Install new gasketing. $Wt = 954.41g \Rightarrow Wt. Pickup = 38.22g \Rightarrow Wt/c Pickup = 4.17 (Yd = 3.36\%)$
	-	-	-	
*	14:20	126 (7)	736.6	Load over; # to top. Over set " <u>46</u> " (50%). Vac. pump on. Argon purge off. Reduce pressure.
(7)	7:20	175	0.4	Vac. pump off. Pressurize w/ argon. Set over at " <u>29</u> " (50%). Leave door open to cool. $Wt = 952.26g \Rightarrow Wt. Pickup = 36.87g \Rightarrow Wt/c Pickup = 3.94 (Yd = 3.17\%)$
	-	-	-	

Comments:

1) After 3 hrs, $\sim 107^\circ C$, atm pressure;
 No evidence of run-out. Set over at "38" (50%). Rotate brick 180° (# to bottom).

2) After 2 hrs, $\sim 128^\circ C$, atm pressure;
 No evidence of run-out. Set over at "46" (50%). Rotate brick 180° (# to top). Remove old door gasketing and replace w/ new gasketing

3) After 17 hrs, $\sim 175^\circ C$, vacuum;
 Note: by 15:30 (7/24/01) the oven temperature was $\sim 160^\circ C$ and the pressure was 0.1 mm \Rightarrow sample dried quickly.
 No evidence of run-out. Set over to "29" (50%) and leave door open to cool.

Comments:

The percent weight yield of the impregnant, insitu, agrees w/ that of the solution in crucibles. Ref. 19J-129-90.

Label 19J-129-89

Performed and Recorded by:

Directed by: J. Levi

Read and Understood by:

Date - / /

Date

Date

Purpose:

To determine the percent yield by weight of solution to compare w/ insitu percent weight yield of VEE c/c composites.

Material:

silicon (IV) oxide, 30% in H_2O , colloidal dispersion (Alfor-Aesent), Lot # A04K09,
0.01 μm particles, in liquid. SA = $320 m^2/g$. Density = 1.20.
Viscosity = 7.7 cps at $82.1^\circ F$, Spec. Grav. = 1.216 at $82.1^\circ F$.

Apparatus:

② 100 ml porcelain. Al foil covers,

Procedure:

Weigh $\sim 10_3$ into each crucible, containing sic boiling chips. Cover w/ Al foil.
Punch holes in Al foil and obtain total weight. Subtract sample weight
to obtain TARE. Processed w/ 195-129-89

Initial Data: (7/24/01)

$$\begin{aligned} 1) \text{ CRUC (CHIPS) + SAMPLE (C)} &= 59.4475 (4) & \text{CRUC (etc) + Al Foil (C)} &= 60.1600 \\ \text{CRUC + CHIPS (C)} &= 49.3229 & \text{CRUC (etc) (C)} &= 59.4975 \\ \text{SAMPLE (C)} &= 10.1746g & \text{Al Foil (C)} &= 0.6625g \Rightarrow \text{TARE} = 49.7854g \end{aligned}$$

$$\begin{aligned} 2) \text{ CRUC + CHIPS + SAMPLE (C)} &= 54.7967 (4) & \text{CRUC (etc) + Al Foil (C)} &= 55.4580 \\ \text{CRUC + CHIPS (C)} &= 44.7620 & \text{CRUC (etc) (C)} &= 54.7967 \\ \text{SAMPLE (C)} &= 10.0347g & \text{Al Foil (C)} &= 0.6613g \Rightarrow \text{TARE} = 45.4233g \end{aligned}$$

Drying Data: (7/24+25/01) - Processed w/ 195-129-89

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:10	108	Atm	Load in oven; in front of Pyrex tray w/ 195-129-89. Purge w/ argon at 5.0 scfh (40%)
-(3)	11:10	106	"	Unload. Cool in desiccator \Rightarrow weigh. Set oven at "58" (50%).
	-	-	-	① CRUC (etc) wt = 54.7662g \Rightarrow wt = 4.7808g \Rightarrow wt Yield = 46.99%
	-	-	-	② CRUC (etc) wt = 49.9430g \Rightarrow wt = 4.5197g \Rightarrow wt Yield = 45.04%
*	11:30	125	Atm	Reload; switch sides. Over set "38" (50%). Purge w/ argon at 5.0 scfh (40%)
-(2)	13:30	130	"	Unload. Cool in desiccator \Rightarrow weigh. Set oven at "46" (50%).
	-	-	-	① CRUC (etc) wt = 53.2721g \Rightarrow wt = 3.2867g \Rightarrow wt Yield = 32.30%
	-	-	-	② CRUC (etc) wt = 48.6710g \Rightarrow wt = 3.2477g \Rightarrow wt Yield = 32.36%

cont'd next page

Performed and Recorded by:

Directed by:

Read and Understood by:

Date

Date

Date

Subject ⁹¹ Drying of ~10g samples of Si. l. wa (II) Oxide, 30% in H₂O, colloidal dispersion (c/c com. app)
 Cross-Reference (if any)

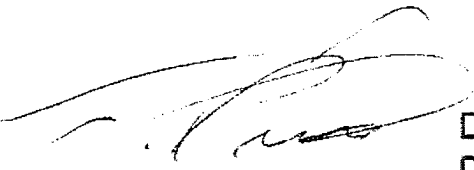
Drying Data (cont'd): (7/24/25/01)

	Time	OVEN TEMP	PRESS (mm)	Comments
*	14:20	126 (°F)	736.6	Reload; reverse sides. Oven set to 126°C. Vac. pump on. Argon purge off.
(17)	7:20	175	0.4	Vac. pump off. Pressurize w/argon. Unload, cool in desiccator. → weigh.
-	-	-	-	① (complete) wt = 53.2182g ⇒ wt = 3.2328g ⇒ wt yield = <u>31.77%</u>
-	-	-	-	② (complete) wt = 48.6127g ⇒ wt = 3.1894g ⇒ wt yield = <u>31.78%</u>

Ave % wt yield = 31.8, $\sigma = 0.01$, $n = 2$

Comments:

Almost exactly the same value for each sample. Same percent weight yield as the insitu % wt yield for 195-129-89.

Performed and Recorded by: 
 Directed by: J. C. Lin
 Read and Understood by:

Date
 Date
 Date

92

Subject VI F 195-129-86 #3-1-B w/ 5.1 wt% (IV) oxide, Colloidal Dispersion (c/c con
Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

- 1) c/c Composite: 195-129-86 #3-1-B; c/c composite via BP process from the 1st block of the 3rd Lawrenceburg trial. 0.25" long K-223-SE pitch fiber and Reiley 105, Load ratio 75/25. No sulfur. Wt (c) = 823.65g, Vol (c) = 554.502 cc, Dens (c) = 1.485 g/cc
- 2) Impregnant: Silicon (IV) oxide, 30% in H₂O, colloidal dispersion. (Alfa-Aesar), Lot #A04K09 0.01 μ m particles, in liquid. SA = 320 m²/g. Density = 1.20. Previous use: 195-129-8 Vis (c) = 7.7 cps at 82.1 °F, S.G. (c) = 1.216 at 82.1 °F.

Apparatus:

Ref. 195-120-10

Procedure:

Ref. 195-129-88

Pump-down Data:

DATE	TIME	PRESS (mTorr)	Comments
7/23	13:15	21	Load block from hot oven (~110 °C, atm)
"	13:25	"	Begin pump-down
"	14:25	45	
7/24	7:10	24	
"	8:25	23	charge traps w/ dry ice-acetone
"	8:55	15	LDR
"	11:45	13	Begin VF

Impregnation Data: (7/23+24/01) - LDR w/ traps charged.

LDR: Initial = 14 mTorr Vis = (7.8 cps at 78.0 °F

5 min = 27 "

10 min = 38 "

15 min = 49 "

S.G. = (1.216) at 78.0 °F

Drop Time = 11:45 (13 mTorr

Unload Time = 7:45 (7/25/01

Held at atmospheric pressure for ~20 hrs.

Comments:

500 ml cylindrical funnel Filled (ie - ~675 ml)

Post Impregnation Data: (7/25/01)

Wt (PV I-1) = 950.80g \Rightarrow Wt Pickup = 127.15g \Rightarrow Wt/o Pickup = 15.44 Vol/o Pickup = 18.86

Performed and Recorded by: 

Directed by: 

Read and Understood by:

Date

Date

Date

Subject VF of 195-129-86 #3-1-B w/ silicosis (X) oxide, Colloidal Dispersion (c/c COMP BP) 93
 Cross-Reference (if any)

Drying Data: (7/25 + 26/01) - Over set "29" (50%). Purge w/ argon at 5.0 SCFH (AIR)

	TIME	OVEN TEMP	PRESS (mm)	Comments
*	8:00	107	Atm	Load over. # to top. Over set "29" (50%). Argon purge 5.0 SCFH (AIR)
(3)	11:00	108	"	Unload over. Over set "38" (50%). Weigh black hot.
	-	-	-	Wt = 907.62g \Rightarrow Wt. Pickup = 83.97g \Rightarrow Wt/o Pickup = 10.19 (Yd = 66.0%)
*	11:20	129	Atm	Load over; # to bottom. Over set "38" (50%). Argon purge 5.0 SCFH (AIR)
(2)	13:20	132	"	Unload over. Over set "46" (50%). Weigh black hot. Install new gasketing.
	-	-	-	Wt = 871.76g \Rightarrow Wt. Pickup = 48.11g \Rightarrow Wt/o Pickup = 5.84 (Yd = 37.8%)
*	13:40	136 (P)	736.9	Load over; # to top. Over set "46" (50%). Vac. pump on. Argon purge off.
(08)	7:40	168	0.4	Vac. pump off. Pressurize w/ argon. Set over at "29" (50%).
	-	-	-	Wt = 863.89g \Rightarrow Wt. Pickup = 40.24g \Rightarrow Wt/o Pickup = 4.89 (Yd = 31.6%)

Comments:

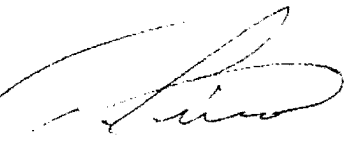
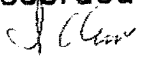
1) After 3 hrs, ~108°C, atm. pressure;
 Rotate brick 180° (# to bottom). Set over at "38" (50%). No evidence of run-out.
 Similar % wt Yrel to 195-129-89 (i.e. 64.1%).

2) After 2 hrs, ~131°C, atm. pressure;
 Scrape off old gasketing and install new, set over to "46" (50%). Rotate
 brick 180° (# to top). No evidence of run-out. H₂O condensed on over door;
 wiped dry.

3) After 18 hrs, ~168°C, vacuum;
 No evidence of run-out. Over set to "29" (50%). Leave door open to cool.

Label 195-129-93

Graph Data: Ref. 195-130-63

Performed and Recorded by: 
 Directed by: 
 Read and Understood by:

Date
 Date
 Date

Purpose:

Ref. 195-129-88

Materials:

Ucle Composite 195-129-86 #3-2-A Ucle composite via BP process. From block #2 of the 2nd Lawrenceburg trial. 0.25" long K-223 SE pitch Fibers. Reilly 155 pitch. Load Ratio = 75% w/o sulfur. Wt wt = 867.13g, Vol wt = 556.247cc, Dens wt = 1.557g/cc
 2) Impregnant: Silica (III) oxide, 30% in H₂O, colloidal dispersion. (Alfa-Aesar), Lot # A04
 0.01um particles, in liquid. SA = 320m²/g. Density = 1.20, Previous use: 7/25/01.
 Viscosity: 7.7 cps at 82.1°F, SG wt = 1.216 at 82.1°F.

Apparatus:

Ref. 195-120-10

Procedure:

Ref. 195-120-15 + 16 * Processed w/ 195-129-86 #3-2-B

Pump Down Data: (7/24/01)

DATE	TIME	PRESS (mm)	Comments
7/24	13:20	19	Load from upper lab
"	13:35	"	Begin pump-down
"	14:35	340	"High" pressure => brick picked up moisture in lab
"	15:55	~205	
7/25	7:20	37	
"	8:35	33(4)	Change traps w/ dry/ice-acetone
"	9:20	22	LDR
"	11:45	18	Begin VI

Impregnation Data: (7/25 + 26/01) - LDR w/ traps changed.

LDR: Initial = 22 m Torr Viscosity = 7.4 cps at 78.0°F Drop Time = 11.45 (16 m Torr)
 5 min = 46 " SG wt = 1.220 at 78.0°F Unload Time = 8.15 (7/26/01)
 10 min = 60 " Held at atmospheric pressure
 15 min = 74 " for ~ 20 1/2 hrs.

Comments:

Filled 500ml cylindrical (Furnel).

Post-Impregnation Data: (7/26/01)

Wt (pvt-1) = 987.85g => Wt Pickup = 120.22g => Wt/O Pickup = 13.86, Vol/O Pickup = 17.

Performed and Recorded by: *[Signature]*

Date

Directed by: *[Signature]*

Date

Read and Understood by:

Date

Subject 1st VI of 195-129-86 #3-2-A w/ silicon dioxide colloidal dispersion (ck COMPAP) 95
 Cross-Reference (if any)

Drying Data: (7/26+27/01) - Over set "29" (50%). Purge w/argon at 5.0 SCFH (AIR)

	TIME	OVEN TEMP	PRESS (mm)	Comments
* (1)	8:30	106	Atm	Load over; # to top. Over set "29" (50%). Argon purge at 5.0 SCFH (AIR)
- (1)	11:30	120	"	Unload over. Over set "38" (50%). Weigh block hot.
	-	-	-	Wt = 944.48g \Rightarrow Wt Pickup = 77.35g \Rightarrow Wt/o Pickup = 8.92 (Yd = 64.3%)
* (2)	11:45	150	Atm	Load over; # to bottom, front of tray. Over set "38" (50%). Argon purge 5.0 SCFH
- (2)	13:45	138	"	Unload over. Over set "46" (50%). Weigh block hot. Install new gasketing.
	-	-	-	Wt = 906.75g \Rightarrow Wt Pickup = 39.62g \Rightarrow Wt/o Pickup = 4.57 (Yd = 33.0%)
* (10)	14:30	176	742.0	Load over; # to top, back of tray. Vac. pump on. Argon purge off. Reduce pressure.
- (10)	8:30	166	0.3	Vac. pump off. Pressurize w/argon. Set over at "29" (50%).
	-	-	-	Wt = 905.02g \Rightarrow Wt Pickup = 37.89g \Rightarrow Wt/o Pickup = 4.37 (Yd = 31.5%)

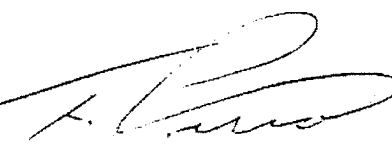
Comments:

- 1) After 3 hrs, ~113°C, atm. pressure;
 No evidence of run-out. Set over at "38" (50%). Weigh block hot. Rotate 180°; # to bottom. Reverse position w/ 195-129-86 #3-2-B (ie. front of Pyrex tray).
- 2) After 2 hrs, ~148°C, atm. pressure;
 No evidence of run-out. Set over at "46" (50%). Weigh block hot. Rotate 180°; # to top. Reverse position w/ "B" (ie. rear of Pyrex tray). Remove old gasketing and install new.
- 3) After 10 hrs, ~171°C, vacuum;
 Set over at "29" (50%). Weigh block hot \Rightarrow reload w/ "B" comparison into VI unit for 2nd impregnation

Comments:

% yield of the impregnant agrees w/ previous 2 blocks; 31.7%, 31.6%, and 31.5%.

Label 195-129-95 #3-2-A 2nd VI: ReF. 195-129-99+100

Performed and Recorded by: 
 Directed by: J. Chris
 Read and Understood by:

Date: / /
 Date
 Date

Subject: VI of 195-129-86 #3-2-B w/ Silicon (II) oxide, colloidal dispersion (c/c comp. as)
 Cross-Reference (if any)

Purpose:

Ref. 195-129-88

Materials:

- 1) c/c composite 195-129-86 #3-2-B (c/c composite via 88 process. From block #2 of the 3rd Lawrenceburg trial, 0.25" long K-223-50 pitch fibers. Reilly 158 pitch. Load ratio = 75/25
 w/o sulfur. Wt (c) = 861.58g, Vol (c) = 571.400cc, Dens (c) = 1.508g/cc
 At impregnant: Silicon (II) oxide, 30% in H₂O, colloidal dispersion. (A) Fu-Aesat, 6+ #A01
 0.01 μ m particles, in liquid. SA = 320 m²/g. Density = 1.20. Previous use: 7/26/01.
 Vis (c) = 7.7 cps at 82.1°F, S.G. (c) = 1.216 at 82.1°F

Apparatus:

Ref. 195-120-15

Procedure:

Ref. 195-120-15016 * Processed w/ 195-129-86 #3-2-A

Pump-Down Data: (7/24/01)

DATE	TIME	PLET (mTorr)	Comments
7/24	13:20	19	Load from open lab w/ #3-2-A
"	13:35	"	Begin pump-down.
"	14:35	390	"High" pressure \Rightarrow blocks picked up moisture in lab.
"	15:55	~205	
7/25	7:20	37(4)	
"	8:35	33(4)	Charge traps w/ dry ice-acetone
"	9:20	22	LDR
"	11:05	18	Begin VI

Impregnation Data: (7/25+26/01) - LDR w/ temp changed

LDR: Initial = 22 mTorr Vis (c) 7.4 cps at 78.0°F Drop Time = 11:45 (18 mTorr)
 5 min = 46 " S.G. (c) = (1.220) at 78.0°F Unload Time = 8:15 (7/26/01)
 10 min = 60 "
 15 min = 74 "

Comments:

Filled 500 ml cylindrical funnel (ie. 467.5 ml).

Post Impregnation Data: (7/26/01)

Wt (pyI-1) = 945.39g \Rightarrow Wt Pickup = 133.81g \Rightarrow Wt Pickup = 15.53 Vol % Pickup = 19.20

Performed and Recorded by: [Signature]

Date:

Directed by: [Signature]

Date

Read and Understood by:

Date

Subject VI of 195-129-86 #3-2-B w/5.1.20(II) oxide, colloidal dispersion (c/c COMPBP)⁹⁷
 Cross-Reference (if any)

Pyrex Data: (7/26 + 27/01) - Over set "29" (50%). Argon purge 5.0 SCFH (AIR). Process w/195-129-95

	TIME	OVEN TEMP	PRESS (MM)	Comments
*	8:30	106	Atm	Load over; # to top. Over set "29" (50%). Argon purge at 5.0 SCFH (AIR).
(3)	11:30	120	"	Unload over. Set over "38" (50%). Weigh block hot.
	-	-	-	Wt = 950.91g \Rightarrow Wt Pickup = 89.33g \Rightarrow Wt/o Pickup = 10.37 (Yd = 66.8%)
*	11:45	150	Atm	Load over; # to bottom, back of tray. Over set "38" (50%). Argon purge 5.0 SCFH.
(2)	13:45	138	"	Unload over. Set over "46" (50%). Weigh block hot. Install new gasketing.
	-	-	-	Wt = 906.48g \Rightarrow Wt Pickup = 44.90g \Rightarrow Wt/o Pickup = 5.21 (Yd = 33.6%)
*	14:30	176	742.0	Load over; # to top, front of tray. Over set "46" (50%). Vac. pump on. Argon purge off.
(18)	8:30	166	0.3	Vac. pump off. Pressurize w/argon. Set over "29" (50%).
	-	-	-	Wt = 703.5g \Rightarrow Wt Pickup = 42.01g \Rightarrow Wt/o Pickup = 4.88 (Yd = 31.4%)

Comments:

1) After 3 hrs, $\sim 113^\circ\text{C}$, atm pressure;
 No evidence of run-out. Set over at "38" (50%). Weigh block hot. Rotate 180° ; # to bottom. Reverse position in Pyrex tray w/195-129-86 #3-2-A (ie. back of tray).

2) After 2 hrs, $\sim 144^\circ\text{C}$, atm pressure;
 No evidence of run-out. Set over at "46" (50%). Weigh block hot. Rotate 180° ; # to top. Reverse position in Pyrex tray (ie. front of tray). Remove old door gasketing and install new.

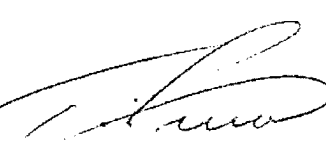
3) After 18 hrs, $\sim 171^\circ\text{C}$, vacuum;
 Set over "29" (50%). Weigh block hot \Rightarrow release w/195-129-95 #3-2-A into VI unit for 2nd impregnation.

Comments:

% yield of impregnant agrees w/previous ③ blocks; 31.7%, 31.6%, and 31.5%.

Label 195-129-97 #3-2-B

2nd VI: Ref. 195-130-01202

Performed and Recorded by: 
 Directed by: J. L. Linn
 Read and Understood by:

Date 7/1/01
 Date 7/1/01
 Date 7/1/01

Subject *Initial Data of Graphitized c/c Composites via BP Process (4th Trial) (c/c Cross-Reference (if any))*

Purpose:

To obtain the initial weights and dimensions prior to vacuum impregnation with "T-143" type phenolic/furfural resin blend for densification.

Materials:

c/c Composites via BP process. Rec'd from P. Sirocky 7/15/01. Two sections, both graphitized. Section "A-1" had one pitch impregnation, section "B-1" did not have a PI. Both graphitized to ~3000°C

Made w/ K-2235E 0.25" long fibers and Kevlar 155 pitch. Load Ratio = 7.5/2 w/o sulfur. Brick 13 of 4th trial.

Procedure:

Essentially same as 195-129-86. Except cooled in desiccator

Initial Data: (7/27/01)

File Path = C:\Program Files\Excel\BP c-c composites\In. Trial.XLS Sheet = BP II

BP C/C COMPOSITES INITIAL WEIGHTS AND DIMENSIONS

Material:

Material: BP-IV-13 A1 and BP-IV-13 B1. Rec'd 7/25/01. Ultrasonic washed 3x for 5 min. in deionized water on 7/26/01. Dimensions were obtained with a Mitutoyo Model CD-8'CS digital caliper. Hot vacuum dried at 124 °C to 0.4 mm pressure from 7/26 to 7/27/01. Weights obtained on Mettler PN 2210 balance on 7/27/01. Note: Both samples have been graphitized to ~3000°C. A1 has one PI. B1 has no PI.

Sample I.D.	Weight (g)	L1 (mm)	L2 (mm)	L3 (mm)	Ave. Length (mm)	W1 (mm)	W2 (mm)	W3 (mm)	Ave. Width (mm)	H1 (mm)	H2 (mm)	H3 (mm)	Ave. Height (mm)	Vol. (cc)	Dens. (g/cc)
4-13-A1	277.03	108.95	108.89	108.98	108.94	93.90	93.92	93.95	93.92	15.65	16.01	16.08	15.91	162.825	1.701
4-13-B1	255.92	107.20	106.19	105.13	106.17	96.81	97.06	97.50	97.12	16.48	16.15	15.65	16.10	165.987	1.542

Dimensioned: 07/27/01
Hot Vac. Dried: 07/26-27/01
Weighed: 07/27/01
N.B. Ref. No. : 195-129-98

Impregnant

4-13-A1 195-129-53 w/50 by vol/o GP5432/Furfural)
4-13-B1 " " " " " / "

N.B. Ref.

195-130-03 #04
195-130-05 #06

Performed and Recorded by:

Directed by: *AC*

Read and Understood by:

Date

Date

Date